

PEEM and NEXAFS studies of hard disks and sliders

Simone Anders¹, Thomas Stammeler¹, C. Singh Bhatia², Joachim Stöhr³,
Walton Fong⁴, Chao-Yuan Chen⁴, David B. Bogy⁴, and C. Heske¹

¹Advanced Light Source, Ernest Orlando Lawrence Berkeley National Laboratory,
University of California, Berkeley, California 94720, USA

²SSD/IBM, 5600 Cottle Road, San Jose, CA 95193

³IBM Almaden Research Center, 650 Harry Road, San Jose, CA 95120

⁴Computer Mechanics Laboratory, Department of Mechanical Engineering,
University of California, Berkeley, CA 94720

INTRODUCTION

X-ray Photo Emission Electron Microscopy (X-PEEM) and Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy are two modern analysis methods based on the availability of tunable X-ray radiation produced by a synchrotron. X-PEEM combines high spatial resolution with the ability to obtain local NEXAFS spectra. NEXAFS yields information about the elemental composition and bonding structure of the elements in the sample, and orientation of molecules (if a preferred orientation is present). All this information is obtained in a PEEM with submicron resolution, in addition to topological contrast which is present in a PEEM also.

Both methods have been applied to study the tribological behavior of the head/disk interface of magnetic storage devices. For storage devices with smaller and smaller spacing between slider and disk approaching pseudo-contact and contact-recording, the tribochemical properties of the system consisting of the carbon overcoat of the disk, the lubricant, and the slider surface (carbon coated or uncoated) become more and more important.

EXPERIMENTAL

Experiments have been performed at the undulator beamline 8.0 of the ALS. The monochromatized X-rays were focused by a Kirkpatrick-Baez pair of mirrors into a 200 μm diameter spot on the sample. The PEEM working principle is based on the electron emission caused by the X-ray illumination of the sample which is proportional to the X-ray absorption. The absorption is a function of the elemental and chemical state of the sample and the X-ray wavelength. An electron optical column forms an image of the emitted electrons. By tuning the X-rays through the absorption edge of the element of interest and taking images at incrementally increased photon energy it is possible to obtain locally resolved NEXAFS spectra with a resolution given by the resolution of the electron optics which is about 200 nm for the microscope we used for the experiments described in this paper. The microscope is a two-lens system operating at a nominal voltage of 10 kV, and it is described in detail elsewhere [1].

RESULTS AND DISCUSSION

Studies of hard disks exposed to thermal desorption experiments

Samples from disks which were coated with 7.5 nm sputter deposited, nitrogenated amorphous hard carbon (CN_x) and lubricated with 0.85 nm perfluoropolyether (Z-dol) were exposed to a thermal desorption experiment. They were heated in UHV (base pressure $<10^{-6}$ Pa) up to 425°C for a duration of 10min. The desorbed gaseous products can be measured using a quadrupole mass spectrometer to determine the processes leading to the degradation of the lubricant due to thermal effects. We have studied the disks before and after the thermal desorption experiments using (not spatially resolved) NEXAFS spectroscopy in the total yield mode. Figure 1 shows the

carbon K edge (1a), fluorine K edge (1b), and nitrogen K edge (1c) spectra before and after heating of the disks.

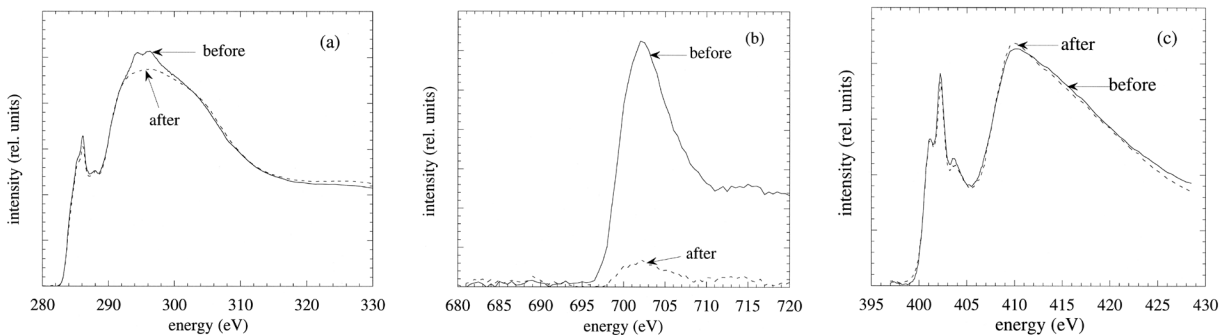


Figure 1: K edge NEXAFS spectra of (a) carbon, (b) fluorine, and (c) nitrogen, of a hard disks before (solid line) and after (dashed line) thermal desorption experiments.

The spectra were normalized to the X-ray beam current so that their relative intensity reflects the relative amount of the corresponding element on the surface of the disk. It can be seen in Figure 1a that the peaks which are characteristic for C=C π^* bonds at 285 eV and σ bonds (broad peak around 300 eV) as well as the C=N peak around 286.2 eV [3] and the C=O peak around 288.1 eV [2] are not influenced by the heat treatment. The peaks at 294.5 and 296.3 eV, respectively, which are due to C-F bonds [4, 5], practically disappear after the heating. The nitrogen spectra (Fig. 1c) show no modification within the accuracy of the measurements, but the fluorine signal (Fig. 1b) is reduced to 10% of its original value by the heating. The data indicate that the CN_x film is not modified in structure and composition by the heating, but 90% of the fluorine from the lubricant has been removed. This is in good agreement to FTIR measurements performed before and after the heating to determine the thickness of the lubricant which showed a thickness of 0.85 nm before and 0.08 nm after heating.

Study of wear tracks on disks

Supersmooth disks were coated with 5 nm cathodic arc deposited amorphous hard carbon and lubricated with 0.85 nm perfluoropolyether (Z-dol). They were used in a tribochamber wear test. The tribochamber is equipped with a spindle in UHV (base pressure $< 10^{-6}$ Pa) and a quadrupole mass spectrometer to measure the erosion products during the wear test. The disks were worn in a continuous drag test with Advanced Air Bearing design sliders at a speed of 0.2 m/s and a load of 30 mN. Two kinds of sliders were used: uncoated sliders ($\text{Al}_2\text{O}_3/\text{TiC}$) and sliders coated with sputter deposited CH_x . After the wear test the disks were studied using the PEEM microscope.

Fig. 2a shows a PEEM image of the edge of the wear track. The scratch is caused by one of the rails of the slider. The image was taken at a photon energy of 280 eV which is below the carbon K edge, therefore the image contrast is mainly topological. Local NEXAFS spectra were taken in the undamaged area of the disk, at the edge of the wear track caused by the rail, and in the area between the rails. Fig. 2b shows the carbon K edge spectra and Fig. 2c the fluorine K edge spectra.

The spectra show that the carbon overcoat/lubricant are identical in the undamaged area and between the rails. At the edge of the wear track in the scratch caused by one of the rails the

fluorine is almost completely removed and the carbon spectrum shows a new peak at 288.5eV which can be attributed to the formation of carboxylic bonds [2].

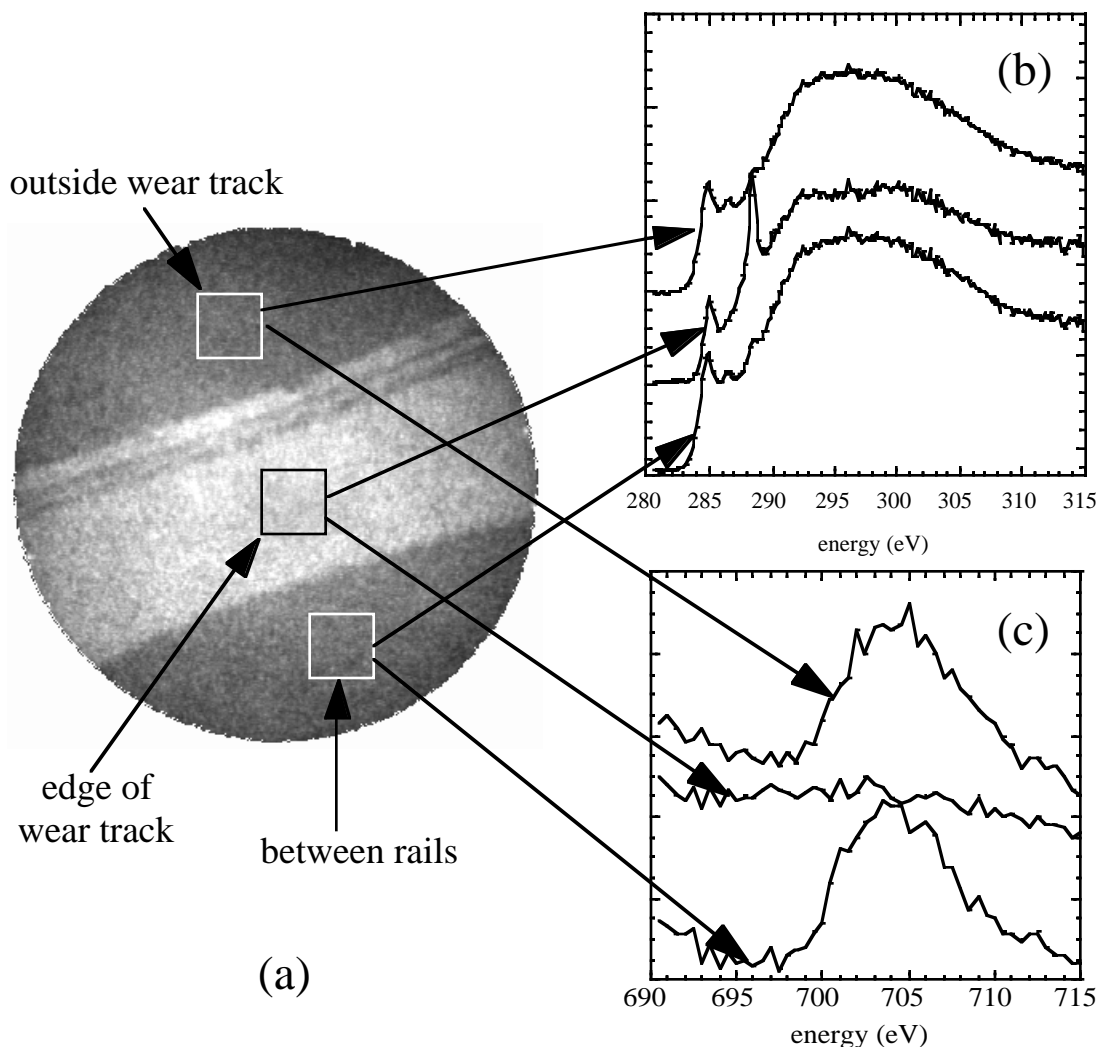


Figure 2: (a) PEEM image of the edge of a wear track taken at a photon energy of 280 eV. The field of view is 150 μ m. (b) Local carbon K edge NEXAFS spectra of indicated areas. (c) Local fluorine K edge NEXAFS spectra of indicated areas.

Study of slider surfaces

The sliders which were used to wear the disks in the tribochamber for the experiments described above were also investigated using the PEEM microscope. We observed a number of scratches at the slider surface of both, the coated and uncoated slider. Figure 3 shows the NEXAFS spectra at the carbon K edge, the oxygen K edge, and the titanium $L_{2,3}$ edge taken in one scratch and outside the scratch of an uncoated slider.

The carbon spectra show basically no carbon on the undisturbed slider surface but a strong signal in the scratch. The spectrum is very similar to the one observed at the edge of the wear track on the disk (Fig. 2b). The oxygen spectrum outside the scratch shows one peak probably connected to the Al_2O_3 of the slider material, but a strong, sharp additional peak around 539 eV appears inside the scratch that can be correlated to the presence of carboxyl bonds [2]. The titanium signal is much weaker in the scratch indicating that some material has been deposited on the surface. All

these data led us to conclude that material has been transferred from the disk to the slider, and this material is the same as the material at the edge of the wear track. It is probably some form of degraded lubricant which is formed during the wear.

On the coated slider we found undamaged CH_x in most of the areas, and observed the same kind of carbon K edge spectrum in scratches with the additional peak around 288.5eV as we found in scratches of uncoated sliders and at the edge of the wear tracks of the disks.

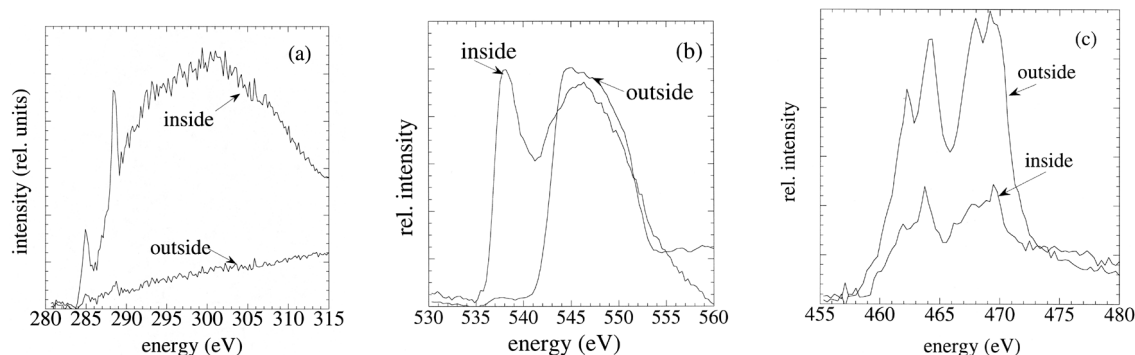


Figure 3: Local NEXAFS spectra at (a) carbon K edge, (b) oxygen K edge, and (c) Ti L_{2,3} edge, taken inside and outside a scratch on a slider surface after wear.

CONCLUSIONS

The experiment which are presented here demonstrate that NEXAFS and PEEM are useful tools to study the problems of the head/disk tribology. It was found that the heating of disks lubricated with perfluoropolyether (Z-dol) up to 425°C for 10min. does not change the CN_x structure and elemental composition of the hard carbon overcoat of the disk, but 90% of the lubricant is removed during this process. Wear of disks lubricated with perfluoropolyether (Z-dol) leads to a strong reduction of the fluorine content in the worn areas and to the formation of new carboxyl bonds. Degraded lubricant showing the carboxyl peak in the carbon K edge spectrum was found also in scratches of coated and uncoated sliders.

REFERENCES

- [1] B. P. Tonner, D. Dunham, T. Droubay, and M. Pauli, *J. Electron Spectrosc. Relat. Phenom.* **84** (1997) 211.
- [2] J. Stöhr, *NEXAFS Spectroscopy*, Springer Verlag, New York, 1992.
- [3] B. Holloway, Thesis, 1997
- [4] Ch. Ziegler, Th. Schedel-Niedrig, G. Beamson, D. T. Clark, W. R. Salaneck, H. Sotobayashi, and A. M. Bradshaw, *Langmuir* **10** (1994) 4399.
- [5] T. Ohta, K. Seki, T. Yokoyama, I. Morisada, and K. Edamatsu, *Physica Scripta* **41** (1990) 150.

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Science Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098. It was also supported by the Computer Mechanics Laboratory, Mechanical Engineering Department, U.C. Berkeley, and by the National Storage Industry Consortium (NSIC).

Principal investigator: Simone Anders, Advanced Light Source, Ernest Orlando Lawrence Berkeley National Laboratory. Email: SAnders@lbl.gov. Telephone: 510-486-5928.